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Applicant: Kyowa Industries Co., Ltd.

Title of Invention: Aerosol Product Using Compressed Gas and Method for  
Producing the Same

Disclosure A: (Column 1, lines 1 to 20)

"[Claims]

[Claim 1] An aerosol product resulting from the process comprising including a stock solution selected from an aqueous solution containing an active component, an aqueous solution containing a surfactant or an alcoholic aqueous solution, a prescribed amount of carbonate or hydrogencarbonate, and a prescribed amount of an organic acid or an inorganic acid in an aerosol vessel, sealing and pressurizing said vessel with an undissolvable compressed gas.

[Claim 2] An aerosol product resulting from the process comprising including a stock solution which is prepared by adding a hydrocarbon oil having carbon atoms of 5 or above to an aqueous solution containing a surfactant, a prescribed amount of carbonate or hydrogencarbonate, and a prescribed amount of an organic acid or an inorganic acid in an aerosol vessel, sealing and compressing said vessel with an undissolvable compressed gas.

[Claim 3] A method for producing the aerosol product according to claims 1 or 2 which comprises including a stock solution, a group of the prescribed amount of carbonate or hydrogencarbonate, and a group of the prescribed amount of an organic acid or an inorganic acid in an aerosol vessel, either of the group of carbonate or the group of acid being added to said stock solution beforehand and another group being added in the form of a water-soluble capsule or the like to said stock solution, and sealing said aerosol vessel."

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**PATENT ABSTRACTS OF JAPAN**

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(21)Application number : **06-238614**      (71)Applicant : **KYOWA KOGYO KK**  
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**(54) AEROSOL PRODUCT USING COMPRESSED GAS AND PRODUCTION OF THE AEROSOL PRODUCT**

**(57)Abstract:**

**PURPOSE:** To obtain the subject product consisting of a specific constitution, CO<sub>2</sub> and an undissolvable compressed gas as propellants, capable of favorably retaining its spraying status to the last by making the best use of the advantages of both the CO<sub>2</sub> as the dissolvable compressed gas and the undissolvable compressed gas.

**CONSTITUTION:** (A) A stock solution, i.e., an aqueous solution or an alcoholic aqueous solution, containing an active ingredient or surfactant, (B) a carbonate or bicarbonate (pref. sodium carbonate or sodium bicarbonate) and (C) an organic or inorganic acid (pref. citric acid or hydrochloric acid), are put into an aerosol vessel followed by sealing and then pressurizing the vessel with an undissolvable compressed gas (e.g. N<sub>2</sub>, helium), thus obtaining the objective product. Specifically, this product is obtained by the following procedure: either of the component B or C is incorporated, in advance, in the component A, and the other is then incorporated in the form of e.g. a water-soluble capsule in the component A. It is recommended that the undissolvable compressed gas be filled immediately after sealing the vessel. It is preferable that the pressure of the gas inside the vessel is adjusted to 3.5-7.8kg/cm<sup>2</sup> at normal temperatures.

## CLAIMS

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[Claim(s)]

[Claim 1] Aerosol products which accommodate and seal organic acid or inorganic acid of an undiluted solution, carbonate of the specified quantity or a hydrogencarbonate, and the specified quantity in an aerosol can by using as an undiluted solution solution having contained an active principle, aqueous liquid containing a surface-active agent, or an alcoholic aqueous solution, pressurize with undissolved compressed gas, and are characterized by things.

[Claim 2] Bubble-like aerosol products which accommodate and seal organic acid or inorganic acid of an undiluted solution, carbonate of the specified quantity or a hydrogencarbonate, and the specified quantity in an aerosol can by using as an undiluted solution liquid which adds a with a carbon numbers of five or more hydrocarbon oil to aqueous liquid having contained a surface-active agent, pressurize with undissolved compressed gas, and are characterized by things.

[Claim 3] It faces accommodating an acid group of a carbonic acid group of an undiluted solution, carbonate of the specified quantity, or a hydrogencarbonate and organic acid of the specified quantity, or inorganic acid in an aerosol can, supplying beforehand either a carbonic acid group or an acid group to an undiluted solution -- already -- a manufacturing method of the aerosol products according to claim 1 or 2 a group of a method of one supplying as a soluble capsule etc., and sealing.

## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to an aerosol product which uses compressed gas as propellants, and a manufacturing method for the same.

[0002]

[Description of the Prior Art] There are what uses a liquefied gas as propellants, and a thing which uses compressed gas in aerosol products. Although, or it is used as a mixture and the characteristic of injection has propane, butane, isobutane, good wood ether, etc. as a liquefied gas, It is an inflammability altogether and a manufacturing process, and the suitable equipment and the management posture which are treated as inflammable high pressure gas are required, and an inflammability and fire cautions are indicated to a product as product precautions for use, and sufficient cautions for transportation storage etc. are required. As compressed gas, carbon dioxide, nitrogen, oxygen, air, helium, argon, etc. are used as propellants. Unlike the time of a liquefied gas, there is a fault of pressure drawdown in this case. That is, when a water-soluble base is used, using carbon dioxide as compressed gas, according to the principle of the gas dissolving of a henry, the carbon dioxide of the quantity of one law dissolves carbon dioxide into a water-soluble base under the pressure of one law. Although the injection state in early stages of a product is good because of this solution gas, an injection state worsens from the middle to a telophase. The capacity of the gas part in a container increases as content fluid decreases in number and this goes, In order for container internal pressure to descend

according to a Boyle's law, and for carbon dioxide to evaporate from a water-soluble base with descent of \*\* and to supply a part of descent pressure, the amount of solution gas in a water-soluble base decreases as a result, and aggravation of an injection state is conjointly caused with descent of a pressure.

[0003]When nitrogen gas is used as compressed gas, unlike carbon dioxide, there is little quantity of the gas dissolving to the inside of liquid. For example, the dissolved amount to the water of nitrogen gas is 0.016ml/mlH<sub>2</sub>O in 20 \*\* and 1atm, and the dissolved amount to the water of carbon dioxide is 0.88ml/mlH<sub>2</sub>O in 20 \*\* and 1atm. Therefore, when it was filled up with a water-soluble base, and it pressurized only with nitrogen gas and was considered as a product, the injection state of the product was bad, in order to improve this, devised valve structure and button structure, and needed the detailed liquid path and the sprayer style so that it could judge from the gas insoluble solution to the inside of liquid. For this reason, there was instability of the jam and spray state which cost starts and also arise from a detailed structure. However, since the Boyle's law was suited surely and the action was carried out to it as a strong point of insolubility compressed gas, it was easy to carry out gas charging, and reduction of the pressure in accordance with consuming content fluid was also able to be foreknown correctly, and could be in charge of the design.

[0004]Thus, soluble compressed gas safer than a liquefied gas and/or the compressed gas of insolubility are used together, As propellants of aerosol products, it depends on the conventional method, and it will depend on a pressure filling method and will fill up with the process of being filled up with two ingredients of compressed gas, using a total of two sets of addressing to inner 1 per ingredient of two ingredients, and pressure restoration heads. When putting soluble compressed gas and insolubility compressed gas together, it is first filled up with soluble compressed gas, and is filled up with insolubility compressed gas after that. In the case of restoration, although the pressure resistance of a can is taken into consideration, filling pressure is set up as highly as possible. This is for, of course making restoration speed quick. Usually, if it says by the pressure like 15 - 20 kg/cm<sup>2</sup>G, it will fill up only with a resisting pressure degree grip of a can, and it is aimed at and \*\* filled up with absorption of liquid in the boiler. In order to bring forward the gas absorption which depends on content fluid, the art which vibrates a can, or devises the mechanism of an aerosol valve and brings a gas transit rate forward is also introduced. However, considerable time is needed when it will say strictly, by the time it makes content fluid absorb carbon dioxide and reaches equilibrium pressure. By the time it carries out pressure restoration of the carbon dioxide at least and is filled up with nitrogen gas, it is necessary to take the idle time for several minutes. This means that the compressed gas of two ingredients cannot be filled up with the process which continued in a series of filling apparatus. Constituting the line which takes the idle time for several minutes and is filled up with the compressed gas of the 2nd ingredient will pay the sacrifice big regarding the place again also as the amount of investment, and it is uneconomical. Although how to mix the compressed gas of two ingredients beforehand as another method, and carry out pressure restoration can be considered, in the case of carbon dioxide and nitrogen gas, it is as physicochemical characteristics.

It comes out. For this reason, a problem arises like a packer. That is, when being filled up, by the time mixed gas results in a filling apparatus through piping, a reducing valve, etc. from the cylinder stored as a raw material, there will be a process of decompression. At this time, the inconvenience which says that liquefaction of carbon dioxide will occur in cooling which depends on adiabatic expansion, and the ingredient ratio of mixed gas will change happens. The mixed gas of compressed gas will become what also has very difficult quality control as material gas and management of the gas volume of the aerosol products which it is complicated and are difficult, and with which it filled up. The above is a big problem in the case of filling up two ingredients with the conventional method as compressed gas.

[0005]

[Problem(s) to be Solved by the Invention]Therefore, the purpose of this invention compensates the fault of the above-mentioned soluble compressed gas (example; carbon dioxide) or undissolved compressed gas (example; nitrogen gas) at \*\*, employs the strong point efficiently, and proposes better aerosol products and bubble-like aerosol products. The purpose of this invention proposes the manufacturing method of the aerosol products which devise the method in which the manufacturing method of the aerosol products which use the binary system compressed gas proposed here is also still more rational than the method currently carried out conventionally, and can be performed simple easily.

[0006]

[Means for Solving the Problem]Composition of following the (1) - (3) can attain the purpose of this invention.

(1) Solution having contained an active principle, aqueous liquid containing a surface-active agent, or an alcoholic aqueous solution is used as an undiluted solution, Aerosol products which accommodate and seal organic acid or inorganic acid of an undiluted solution, carbonate of the specified quantity or a hydrogencarbonate, and the specified quantity in an aerosol can, pressurize with undissolved compressed gas, and are characterized by things.

(2) Liquid which adds a with a carbon numbers of five or more hydrocarbon oil to aqueous liquid having contained a surface-active agent is used as an undiluted solution, Bubble-like aerosol products which accommodate and seal organic acid or inorganic acid of an undiluted solution, carbonate of the specified quantity or a hydrogencarbonate, and the specified quantity in an aerosol can, pressurize with undissolved compressed gas, and are characterized by things.

(3) It faces accommodating an acid group of a carbonic acid group of an undiluted solution, carbonate of the specified quantity, or a hydrogencarbonate and organic acid of the specified quantity, or inorganic acid in an aerosol can, supplying beforehand either a carbonic acid group or an acid group to an undiluted solution -- already -- a manufacturing method of the aerosol products according to claim 1 or 2 a group of a method of one supplying as a soluble capsule etc., and sealing.

[0007]It is this invention's accommodating and sealing an undiluted solution, carbonate or a hydrogencarbonate (carbonic acid group) and organic acid, or inorganic acid (acid group), and pressurizing by gas of compression insolubility, By considering it as a system which good aerosol products of an injection state are obtained [ system ] to the last, and generates carbon dioxide from a carbonic acid group and an acid group. A manufacturing

method of aerosol products which can perform easily restoration, gas (for example, carbon dioxide) of compression solubility and gas (for example, nitrogen gas) of undissolved compression (it may be called compression insolubility), of two ingredients simple can be provided. Even when not using gas of compression insolubility depending on a kind of product of aerosol, it is generating carbon dioxide from carbonate or a hydrogencarbonate, organic acid, or inorganic acid in an aerosol can, A manufacturing method of good aerosol products of an injection state and aerosol products which can be performed simple easily can be provided to the last. When the pH of mixed liquor is mostly adjusted to neutrality, the specified quantity of a carbonic acid group and an acid group becomes as calculation by a chemical equation, and is preferred. If what enclosed organic acid or inorganic acid with a water-soluble capsule is added, even if it adds into liquid in which carbonate or a hydrogencarbonate was dissolved, a reaction will not occur promptly but it will become easy to do seal work. Depending on the case, organic acid or inorganic acid can also be used as powdered. In this invention, although an undiluted solution in particular is not limited, what constitutes aerosol products, such as solution containing an active principle, solution containing a surface-active agent, or an alcoholic aqueous solution, is preferred. In this invention, an undiluted solution uses as an undiluted solution what constitutes aerosol products of the shape of a bubble as for which five or more carbon numbers added a hydrocarbon oil of 5-7 preferably in water solubility having contained a surface-active agent. As long as ingredients in particular, such as the above-mentioned active principle, a surface-active agent, and an alcoholic aqueous solution, are not limited but are used for aerosol products, any may be sufficient as them.

[0008] This invention is explained in detail below. Carbon dioxide is first generated in an aerosol can. namely, -- dissolving carbonate or a hydrogencarbonate into solution, emulsion liquid, or an alcoholic aqueous solution having contained various active principles, and accommodating this in an aerosol can -- this -- organic acid or inorganic acid -- \*\* -- [ a fixed quantity ], if it seals, generating of a constant rate of carbon dioxide will be seen. In this invention, the specified quantity means quantity which was computed by chemical equation and determined from the amount of carbon dioxide which you want to generate. About generating of this carbon dioxide, sodium bicarbonate is taken, for example as a hydrogencarbonate, and citrate is taken and explained as organic acid. Sodium bicarbonate is chemical formula  $\text{NaHCO}_3$  and citrate is  $\text{C}_6\text{H}_8\text{O}_7$  and  $\text{H}_2\text{O}$ . A reaction in these two persons' solution generates 3-mol carbon dioxide, 1 mol of citrate soda, and 3-mol water in 1 mol of citrate, and 3 mol of  $\text{NaHCO}_3$  as known well. That is, it is a formula when a chemical equation expresses. [1] It becomes.

[0009]

[Formula 1]

[0010] Each molecular weight is citrate = 210.14  $\text{NaHCO}_3$  = 84.02  $\text{CO}_2$  = 44, and according to the amount of carbon dioxide which you want to generate, the quantity of required  $\text{NaHCO}_3$  and citrate depends on calculation, and can be calculated. On the other hand, the dissolved amount of the carbon dioxide to an aqueous solution depends on a variety of

research, and is grasped correctly. For example, if it depends on books "soft drinks" (Korin issue soft-drinks member-of-editorial-board meeting), at the temperature of 25 \*\*, the carbon dioxide absorption index in each pressure is indicated about sucrose solution (768-769 pages).

[0011]

[Table 1]

[0012]The carbon dioxide mass C is  $C=(V) \times (\text{absorption index}) \times 1.976 \text{ (gr/l)}$  here....

Formula (2)

It comes out and asks.

however -- Volume (l) of V= solution

It is as follows, when take an aerosol can (fullness of 156 ml) now, this is filled up with 90 ml of undiluted solutions (namely, solution having contained an active principle) of this example (1), and it is filled up with carbon dioxide which depends on the case, and is made internal pressure <sup>2</sup> of 5.0kg/cm, and it depends on a formula (2) and the required amount of carbon dioxide is calculated. [Table 1] An absorption index of carbon dioxide is the carbon dioxide mass (it is considered as C<sub>1</sub>) in at 25 \*\* and absorption-index =4.43 solution more.

[0013]

[Expression 1]

[0014]Since the carbon dioxide which occupies 66 ml of C<sub>1</sub>=0.787gr head spaces also shows the pressure of 5 kg/cm<sup>2</sup>, it is the carbon dioxide mass (it is considered as C<sub>2</sub> gr) in this space.

[0015]

[Expression 2]

[0016]The carbon dioxide gross weight in a  $C_2=0.760\text{gr}$  aerosol can is  $C=C_1+C_2=1.547$  (gr).

The above-mentioned chemical equation For obtaining  $\text{CO}_2$  of the above-mentioned 1.547gr since 3-mol carbon dioxide is generated from 3-mol  $\text{NaHCO}_3$  and 1 mol of citrate if it depends on [1] [0017]  
[Expression 3]

[0018]  
[Expression 4]

[0019]  
 $\text{NaHCO}_3$  .... 2.954gr .... [Expression 3] It is citrate more..... 2.462gr .... [Expression 4]  
The amount of each of a twist is needed. This quantity is the specified quantity in each. If it depends on this example (back appearance), it will be an aerosol can (fullness of 156 ml).

Solution 90ml $\text{NaHCO}_3$ 3gr citrate It depended on the formula of 2.5gr and the result of internal pressure  $5 \text{ kg/cm}^2$  has been obtained. That is, 90 ml of the above-mentioned solution is taken, and 3-g  $\text{NaHCO}_3$  is put in and stirred to this. The aerosol can was filled up with this and the internal pressure of  $5 \text{ kg/cm}^2$  (room temperature) was obtained the place which put in the citrate of 2.5gr wrapped in wafer paper paper, and carried out crimp of the aerosol valve, and 7 minutes afterward.

[0020]If citrate is directly thrown into liquid when making undiluted solutions, such as solution having contained various active principles, carry out the specified quantity dissolution of the  $\text{NaHCO}_3$  in the process of the above pressure generating and adding the citrate of the specified quantity to this, A reaction occurs promptly, a foaming phenomenon is caused, and since content fluid overflows outside a container mouth part, the work which carries out crimp of the valve cannot be performed. therefore, a water-soluble resin Ex polyvinyl alcohol film -- water solubility is not carried out -- it is -- etc. - - it is good to wrap a constant rate of citrate, to make it the shape of a capsule, and to add. A constant rate is tableted and a periphery is made to permeate the powder (silica) of coating spherical porosity with water soluble resin as a method of making it into the shape of a capsule. It puts into the semi-spherical hollow capsule which fabricated the periphery in the coat and the water-soluble range if needed, and two pieces are doubled and it pastes up. moreover -- it being important to adjust the pH of solution to neutral vicinity, before dissolving carbonate or a hydrogencarbonate in the solution having contained various active principles in the meaning which manages the yield of carbon dioxide correctly, and setting to this invention -- pH 5-8 -- it is pH 6.0-7.9 preferably. as the adjustment method of pH -- as the regulator by the side of acidity -- organic acid and inorganic acid (an example.) On the other hand, triethanolamine, caustic alkali of sodium,



caustic potash, diethanolamine, an ammonia solution, a borax, monoethanolamine, etc. are used as a regulator by the side of alkali using citrate, tartaric acid, chloride, etc.

[0021]As explained above, soluble compressed gas can be generated in an aerosol can. In this invention, it is further filled up with the compressed gas of insolubility in an aerosol can. One copy of gas which the pressure in a can decreases as space volume will increase if aerosol contents are consumed when only soluble compressed gas is propellants, and is dissolving this in contents simultaneously with it evaporates. A state (for example, the state of a spray state, a spray pattern, and a bubble, spouting force) when injecting as a result changes to the worse one. If filled up with insolubility compressed gas, it can prevent soluble compressed gas re-evaporating within an aerosol can, and a state when also preventing and injecting descent of \*\*\*\* internal pressure can be maintained at a good state. However, the product can use it to the last comparatively good by the injection which depends only on carbon dioxide. In that case, it produces commercially using generating of the carbon dioxide which depends on this invention, and it can finish without using insolubility compressed gas. However, it is thought preferred to already use beam insolubility compressed gas together theoretically. It is economical in it being only carbon dioxide.

[0022]As compressed gas of insolubility, nitrogen gas is the typical thing, for example. It can be filled up with restoration of nitrogen gas even if it depends on the pressure filling-up method currently enforced from the former in the industry, or a under cup method. However, since there is a pressure which carbon dioxide generates at the process with which it was filled up previously, the filling pressure of nitrogen gas will be filled up with the pressure which balanced the last setting pressure and carbon-dioxide-pressure power. If it depends on an experiment [Nitrogen filling setting pressure] = [The last setting pressure] - [Carbon-dioxide-pressure power]

It may think. Namely, when the pressure of the carbon dioxide filled up with the method explained previously is [ and ]  $5 \text{ kg/cm}^2$  to make the pressure of aerosol products into 25 \*\* and  $7 \text{ kg/cm}^2$  [Nitrogen filling setting pressure] =  $[7] - [5] = 2 \text{ kg/cm}^2$  (at 25 \*\*) may be sufficient. Being filled up with nitrogen after the carbon dioxide with which it was filled up previously shows  $5 \text{ kg/cm}^2$  as a practical question, It is also a reason with the operation effect which carries out nitrogen gas restoration by there being a problem also in pressure immediately after the injection of two ingredients for carbon dioxide generating finishing, and completing seal work since a management top is also disadvantageous, and is explained later. Here, immediately after completing seal work, it puts immediately after completing seal work, and for [ 0 second - ] 5 seconds is specifically shown.

[0023]Next, it explains to the generating process of carbon dioxide in detail. The chemical equation of the above [ generating of carbon dioxide ] [I] It is as being shown. Generating of carbon dioxide was tested using the materials of the following size like the experiment of the place explained above. Namely, \*\* Vessel \*\* Quantity 156-ml aqueous liquid 90-ml head space Place performed by calculating and changing 66 ml of quantity of  $\text{NaHCO}_3$  and citrate here according to quantity calculation of a reaction formula The graph shown in [drawing 1] was obtained. That is, it is saying that it depends on controlling the quantity of  $\text{NaHCO}_3$  and citrate, and pressure generating in a can can be controlled well. When depending on the conventional method and carrying out pressure restoration of the carbon dioxide, it must work in consideration of various

factors, such as the number of carbon dioxide absorbent system of content fluid, a rate of absorption, restoration speed, filling pressure, pressure resistance of a can, a mechanism of an aerosol valve, contents solution temperature, and carbon dioxide temperature, having to carry out condition adjustment at the time of restoration each time. (b) which dissolves the injection of  $\text{NaHCO}_3$  in (a) aqueous liquid at fixed percent when depending on this invention -- among carrying out liquid restoration as solution of the (c) fixed concentration which is measured beforehand and used as a soluble capsule etc., A suitable method can be adopted, the same method as (a), (b), and (c) can be taken about citrate again, and a rational and easy process can be chosen combining these.

(a) If it carries out, it can carry out as batch work. Namely, what is necessary is to accommodate the solution having contained the active principle in the big tank, to put predetermined  $\text{NaHCO}_3$  into this and just to carry out mixed stirring. However, since obstacles, like the retention period of solution becomes short may be encountered according to this work, it is necessary to investigate a priori.

(b) Since it becomes the addition work at the time of restoration, it is easy to automate as work. However, there is a problem (deterioration, moisture absorption) of the prolonged storage of a capsule.

(c) Since it can carry out as batch work, it is rational, but volume management at the time of filling work must be performed strictly.

If  $\text{NaHCO}_3$  is performed by the method of (a) and takes the post of citrate, it is preferred to carry out by encapsulating by the method of (b). When this reverse is performed, it is not impossible, but it is not desirable as a process. That is, citrate is put into solution. [(a) a method] When the can was filled up and  $\text{NaHCO}_3$  is added to this [(b) a method] Although a regular pressure is generated, it worries about the influence which the solution into which citrate was put becomes low about with three temporarily and to which pH gives it to the whole solution of this. However, pH of the product which added  $\text{NaHCO}_3$  and was completed in this case is the 6.7th place, and is normal.

[0024] Although in particular carbonate, the hydrogencarbonate, organic acid, and inorganic acid that can be used by this invention are not limited, what is shown below is preferred. As carbonate, sodium carbonate ( $\text{NaHCO}_3$ ), calcium carbonate ( $\text{CaCO}_3$ ), Magnesium carbonate ( $\text{MgCO}_3$ ), rubidium carbonate ( $\text{Rb}_2\text{CO}_3$ ), potassium carbonate ( $\text{K}_2\text{CO}_3$ ), and cesium carbonate ( $\text{Cs}_2\text{CO}_3$ ) are mentioned. As a hydrogencarbonate, sodium bicarbonate ( $\text{NaHCO}_3$ ), potassium bicarbonate ( $\text{KHCO}_3$ ), ammonium acid carbonate ( $\text{NH}_4\text{HCO}_3$ ), and aerated water matter lithium ( $\text{LiHCO}_3$ ) are mentioned. As organic acid, citrate, acetic acid, and tartaric acid are mentioned and chloride and sulfuric acid are mentioned as inorganic acid.

[0025] Next, the packer of insolubility compressed gas degree is explained in detail. If it attaches like the packer of insolubility compressed gas, for example, nitrogen gas, as explained above, it can fill up with the conventional pressure filling method. However, generally it is necessary to take into consideration various factors, such as the pressure in a can, restoration speed, filling pressure, pressure resistance of a can, an aerosol valve mechanism, and gas temperature, carries out in the difficulty of restoration after carbon dioxide, and is that of a potato. However, when depending on this invention, after the ingredient injection for carbon dioxide generating, and container seal, since nitrogen gas restoration is performed promptly, the pressure in a can is also close to abbreviated atmospheric pressure, and since low pressure may be comparatively sufficient also as

setting out of the can internal pressure which depends on a nitrogen component, filling work is stabilized and it can work comfortably. Nitrogen, helium, argon, neon, and oxygen can be used as gas of compression insolubility used by this invention. The pressure in an aerosol can depends on 8th section of security rule the 67th paragraph etc., such as 12th section of general high pressure gas security rule the 27th paragraph, and industrial complex, and the internal pressure is regulated at the temperature of 35 °C that it is below 8 kg/cm<sup>2</sup>, and needs to follow it. It seems that it is good to design to 3.5 kg/cm<sup>2</sup> - 7.8 kg/cm<sup>2</sup> at ordinary temperature preferably in order to maintain the injection state of a product good.

[0026]Below, the comparative experiments of the compressed gas of this invention and the things other than this invention were conducted below, and they were evaluated.

(Comparative experiments)

Drawing 2 is AL container (fullness of 156 ml).

Purified water 90mlNaHCO<sub>3</sub>3g citrate It was filled up with 2.5 g like the following (a) - (c), and the pressure reduction accompanying contents consumption was tested after neglect on the 1st. The result It collected into the graph shown in [drawing 2].

(\*\*) Two ingredients (that in which nitrogen carried out application-of-pressure restoration 2 kg/cm<sup>2</sup> promptly after container sealing)

(\*\*) Reaction products (what adds and seals NaHCO<sub>3</sub> and citrate similarly to (\*\*), and is not filled up with nitrogen)

(\*\*) a pressurized product (crimp of 90 ml of the purified water is put in and carried out, and 5 kg/cm<sup>2</sup> is pressurized by the conventional method) (NaHCO<sub>3</sub> and citrate are not used -- evaporating :liquefied carbon dioxide and decompressing -- an aerosol gas charging machine -- an aerosol pipe -- pressure restoration -- carrying out (pressure filling method) -- it carries out)

The residue (0 to 100%) of the liquid in a can and the relation of can internal pressure are expressed about each product (\*\*), (\*\*), and (\*\*). When it depended on this, as for pressurized product (c), can internal pressure descended from 5 kg/cm<sup>2</sup> to 4.3 kg/cm<sup>2</sup> one day after after restoration. Descent of a pressure is intense when content fluid passes over 20% of a residue. As compared with this, a pressure changes reaction product (b) in not a day. This is considered that gas had fully been saturated from the beginning since carbon dioxide reacted and was made in the homogeneous system of content fluid. the pressure drawdown accompanying content fluid reduction -- abbreviated -- it is uniform. Two-ingredient product (a) was what carried out nitrogen application of pressure promptly after container sealing, and, for the reason, was the initial pressure of 7.3 kg/cm<sup>2</sup> also after one day. Even if the liquid in a container became zero in this case, in more than 2 kg/cm<sup>2</sup>, internal pressure was maintaining \*\*\*\* and normal injection capability.

Although this invention proposes the pressurizing method in two ingredients, it is considered that it can also carry out the product of only carbon dioxide as a part of case like the (b) products which depend on this experiment.

[0027]Next, when the same examination products as the above-mentioned (a), (b), and (c) were made, inner capacity was injected and it went, the amount of evaporation of the atmosphere which catches the injected liquid to a beaker and evaporates from there was measured. The result It was shown in [drawing 3]. It can be surmised that this amount of evaporation (percent) is a number proportional to the quantity of the carbon dioxide contained in content fluid. They will be (a) products if it depends on this result. -- The

evaporation rate of carbon dioxide is stable in approximately regulated.

(b) Product -- The evaporation rate is comparatively stable.

(c) Product -- An evaporation rate is large at first, it follows on content fluid decreasing in number and going, and an evaporation rate decreases.

it is shown under a table -- as -- Spray state (b) products with the sufficient state to the (a) product -- last of a spray -- about 10% of a residue -- until -- Good spray state (c) products -- a spray state worsens at least with 30% of a residue -- it mentioned above -- as -- the propellants of actual condition aerosol products -- LPG, DME or those things that were blended are most, and all are combustible gas. That is, detailed cautions and management are required of each stage of a manufacturing facility, manufacturing control, circulation, storage, and consumption. Since safe and harmless compressed gas (for example, nitrogen gas, carbon dioxide) is used as an ingredient by incombustibility when this depends on this invention, it becomes what has several safe steps. Since \*\* also serves as the undiluted solutions with main water-soluble undiluted solution, emulsion liquid, etc., it can manufacture as nonflammable products synthetically as a product.

[0028] Since carbon dioxide has bacteriostatic action, it is said that what was pressurized now does not have contamination of a microorganism. However, therefore, the pH of aqueous liquid changes to the pressurized pressure. That is, by the pressure more than 3.5 kg/cm<sup>2</sup>, it is said that pH of water becomes four or less. The quality top of a product of the aqueous undiluted solution of aerosol receiving an unusual descent of pH for carbon dioxide is not good. The case is the best for making nitrogen gas play the role of the propellant for pressing down low the application of pressure which depends on carbon dioxide for this reason (example; 2 kg/cm<sup>2</sup>), lessening descent of pH, and injecting. In a conventional method, a space is also widely needed, and being filled up with two ingredients (solubility and undissolved) of compressed gas as explained above has the very disadvantageous economical amount of an investment greatly. When depending on this invention, production that it is dramatically economical and \*\* is also safe and exact can be performed. When requiring only one ingredient of carbon dioxide, it becomes unnecessary [ the manufacturing facility, piping, etc. of high pressure gas ], and easy, safe, and economical production is attained.

[0029]

[Example]

(Example 1)

Mixed liquid (face toilet)

1, three butylene glycols Two Weight % oleyl alcohol 0.1 \*\* POE(20) sorbitan mono-laurate ester 0.5 \*\* ethanol 10.0 \*\* scent Charge \*\* Quantity Paraben \*\* Quantity Purified water The above-mentioned mixed liquid 86.9% of the weight. 90 ml is put into the aluminum can for aerosol (capacity of 156 ml), and NaHCO<sub>3</sub> 3g is measured and supplied to this. Prepare the capsule which took 2.5 g of citrate independently and was packed with the film of polyvinyl alcohol, supply this, and crimp of the valve is carried out promptly. Immediately after that, nitrogen gas was filled up with 2 kg/cm<sup>2</sup>G (application-of-pressure restoration is carried out), the product showed the can internal pressure of 7 kg/cm<sup>2</sup> at 25 \*\* 10 minutes afterward, and the injection situation with a good time of equipping with and injecting a top button was shown. The internal pressure of the can after injecting the whole quantity for inner capacity in the good state showed the residual pressure of 2 kg/cm<sup>2</sup>.

[0030](Example 2)

Mixed liquid (alcohol for disinfection / sterilization)

Propylene glycol 1.0 % of the weight Irgasan 0.5 \*\* ethyl alcohol 95% 72.0 \*\* purified water 90 ml of the 26.5 \*\* above-mentioned mixed liquid is put into the aluminum can for aerosol (capacity of 156 ml), and  $\text{NaHCO}_3$  2.5g is measured and supplied to this.

What carried out the mixture solution by this ratio beforehand on the occasion of preparation of mixed liquid may be put into a 92.5g container. The capsule which accommodated 2.1 g of citrate wrapped in the water soluble film in this is thrown in, and  $3 \text{ kg/cm}^2$  application-of-pressure restoration of the nitrogen gas is promptly carried out for a valve after crimp. In about 10 minutes, product internal pressure shows  $7.1 \text{ kg/cm}^2$  and shows a good injection state. When the whole quantity could be injected in the state of good injection and content fluid was exhausted, can internal pressure was  $2 \text{ kg/cm}^2$ .

[0031](Example 3)

Mixed liquid (face toilet)

0.5% of hyaluronic acid liquid 0.5 % of the weight Oleyl alcohol 0.2 \*\*\*\* seaweed extract 0.1 \*\*\*\* purified water 90 ml of the 99.2 \*\*\*\* above-mentioned mixed liquid is put into the same container as Example 1,  $\text{NaHCO}_3$  4g is supplied to this, the soluble capsule which included 3.3 g of citrate is added, and crimp of the valve is carried out. A product shows the internal pressure of  $6.7 \text{ kg/cm}^2$  in 10 minutes, and its injection state is also good.

[0032](Example 4)

Mixed liquid (mousse for the hairs)

propylparaben 0.1 % of the weight Methylparaben . 0.1 \*\*\*\* ultraviolet inhibitor 0.3 \*\*\*\* ethanol 99%. 10.0 \*\*\*\* scent Charge 0.1 \*\*\*\* purified water 78.7 \*\*\*\*

polyoxyethylene cetyl ether 1.2 \*\*\*\* butyl methacrylate copolymer 7.0 \*\*\*\* dipropylene glycol 1.5 \*\*\*\* silicone The 1.0 \*\*\*\* above-mentioned mixed liquid. 90 ml is put into an aluminum container (capacity of 156 ml).  $\text{NaHCO}_3$  2gr is put into this. Or mixed liquid mixed into this ratio may be put in 92 gr. The soluble capsule and isopentane 5gr which included citrate 1.7gr are put into this, crimp of the valve is carried out promptly, and application-of-pressure restoration is carried out so that it may become the internal pressure of  $3 \text{ kg/cm}^2$  about nitrogen gas just behind that. A product presents the internal pressure of  $6.6 \text{ kg/cm}^2$  in 10 minutes, a bubble with a good time of depending on an adapter (spout) and injecting is injected, and the bubble is maintained to fitness for more than 2 minutes. Simultaneously with generating of carbon dioxide, it is citrate soda so that it may see in the letter which indicated the chemical formula etc. in this case.

It \*\*\*\*. This substance is useful as a raw material for cosmetics, and is widely used for makeup cosmetics, basic cosmetics, etc. It indicates in 140 pages of cosmetic material dictionaries (daylight chemicals).

[0033]

[Effect of the Invention] This invention has the following effects so that it may become

clear by having stated to the above example. Application-of-pressure restoration of the insolubility compressed gas is carried out at the same time it puts in and closes a hydrogencarbonate, organic acid, or inorganic acid to the solution, the emulsion liquid, and the alcoholic aqueous solution having contained various active principles, and the aerosol products which have two ingredients, soluble compressed gas and insolubility compressed gas, can be produced rationally. The aerosol products which have such two ingredients were not able to be mass-produced with the conventional production method. The aerosol products of the compressed gas application of pressure proposed to this invention were developed under the novel idea which combined the advantage which soluble compressed gas has, and the advantage which insolubility compressed gas has. Although the atmosphere which melted when it has the character which melts into content fluid and was injected from the aerosol can expands rapidly, is subdivided, soluble compressed gas subdivides contents simultaneously and a good spray state is shown. It follows on contents being injected on the other hand and decreasing, and there is reduction of the pressure accompanying increase of space (head space), and the gas which is carrying out \*\* ON into liquid evaporates in order to compensate this. As a result, there is a tendency for the gas volume in liquid to decrease and for a spray state to get worse. On the other hand, since insolubility compressed gas does not melt into content fluid, there is no effect of the expansion and fragmentation at the time of injection, but since it is not injected out of a container with liquid at the time of injection of content fluid, the effective operation as propellant is maintained, without losing the gas volume with which it filled up at first. It depends on combining the two above-mentioned ingredients, and the fragmentation effect of the jetting liquid which maintains can internal pressure properly and depends on a pressure is also made to maintain taking advantage of the feature of insolubility gas in this invention at the same time it presses down the evaporation to the head space of soluble compressed gas and makes the good effect in the case of injection maintain. With the character of a product, insolubility compressed gas may be made unnecessary and it can produce commercially only with the carbon dioxide emitted by this invention in that case. When filled up only with carbon dioxide, when depending on the conventional pressure filling-up method, filling pressure of the problem 2 carbon dioxide of the reliquefaction in piping for 1 carbon-dioxide conveyance is not made more than a certain constant pressure (for example, 20 kg/cm<sup>2</sup>), and, This problem will be solved if they surely depend on this invention, although shake of an aerosol can and fixed time (for example, 20 to 30 seconds) had a difficult problem of restoration by necessity etc. by gas saturation.

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[Translation done.]